

PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Process for Manufacturing Phenol and Acetone from Cumene Hydro-Peroxide

5 We, SOCIETA ITALIANA RESINE, an Italian Body Corporate of 33, Via Grazioli, Milan, Italy, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a process for producing phenol and acetone from cumene hydroperoxide.

15 In Specification No. 684,039 there is described and claimed a process in which cumene hydroperoxide is decomposed in a liquid reaction medium comprising concentrated sulphuric acid and acetone. More particularly, cumene hydroperoxide is gradually added to a solution of acetone and 0.05—10% by weight of concentrated sulphuric acid at a temperature between 0° C. and 20 100° C.; upon completion of the decomposition, the sulphuric acid in the reaction mixture is neutralized and the mixture then distilled to recover phenol.

25 Since in the process mentioned above large volumes of acetone are involved which require bulky apparatus, a correspondingly large consumption of heat and losses of acetone, it is an object of the present invention to provide a process for producing 30 phenol and acetone from cumene hydroperoxide wherein the use of acetone is advantageously avoided. It is a further object of the invention to substantially limit the volume of the reaction mixture (as compared with the prior process mentioned above) with 35 a view to obtaining a better temperature control of the mixture and a reduction in the consumption of sulphuric acid and respective neutralizing agent; in the prior process 40 relatively large volumes of concentrated sulphuric acid are necessary on account of the presence of acetone which dilutes the acid in the reaction medium.

45 According to the present invention, there is provided a process for producing phenol and acetone from cumene hydroperoxide by decomposing the latter by means of concentrated sulphuric acid, the process being characterized in that a solution of the cumene hydroperoxide in cumene at a concentration 50 of at least 70% and concentrated sulphuric acid are gradually added to cumene or to a previously decomposed solution of cumene hydroperoxide in cumene, the rate of addition being so controlled that the concentration 55 of the acid in the reaction mixture is maintained in the range of from 0.1% to 1.1% by weight throughout the process. The reaction temperature is preferably in the range of from 40° to 100° C. 60

The process differs substantially from the prior processes mentioned above. For example, no acetone is added to the reaction mixture to provide a common solvent for the sulphuric acid, cumene, cumene hydroperoxide and phenol, the only acetone present 65 being that obtained in the course of the decomposition reaction. According to the prior processes, a large proportion of acetone must be provided in a reaction vessel both initially and throughout the reaction in order to 70 maintain the reaction medium homogeneous; on the contrary, in the process of the present invention no provision of addition of acetone is made to that end. In one embodiment of 75 the process, a volume of cumene is provided in a reaction vessel, whereupon cumene hydroperoxide dissolved in cumene and sulphuric acid are added while vigorously stirring. Alternatively, a volume of previously 80 decomposed cumene hydroperoxide dissolved in cumene can be substituted for the above-mentioned volume of cumene in the vessel.

85 When the addition of cumene hydroperoxide and acid to the reaction vessel is completed, the reaction mixture can be discharged

and conveyed, after neutralisation, to a distillation apparatus.

The problem of recovering and recycling the sulphuric acid does not arise, for the acid can be neutralized each time without detrimentally affecting the economy of the process, since the quantity of acid employed in the process and subsequently neutralized does not exceed the quantity of acid lost in recycling the large quantities of acid employed in the prior processes. This invention therefore dispenses with the extremely expensive apparatus which was necessary with prior processes for recovering and recycling sulphuric acid.

The following examples illustrate the invention.

EXAMPLE 1

A standard 18—8 stainless steel autoclave, with a capacity of 120 litres and equipped with a Vibromixer stirrer, reflux cooler, heating and cooling coil, was filled with 20 litres of pure cumene containing 1% by weight of concentrated sulphuric acid. The liquid was heated to 50° C. with stirring, and the water cooling the autoclave was adjusted to a temperature of about 30° C. and a delivery of 200 litres per hour. A 75% solution of cumene hydro-peroxide in cumene together with sulphuric acid were then added to the liquid in the autoclave.

The sulphuric acid was supplied in such quantity as to maintain a constant concentration of 1% with respect to the hydro-peroxide solution.

The addition of sulphuric acid may be conveniently effected slightly in advance of the hydro-peroxide solution. However, the sulphuric acid concentration in the reaction mixture should not be allowed to increase to over 1.1%.

In this example, a total quantity of about 80 kgs. of cumene hydro-peroxide solution were added to the liquid in the autoclave within two hours. When the addition was completed, the reaction mixture was cooled to about 25° C. The necessary quantity of anhydrous sodium carbonate or precipitated calcium carbonate was added to neutralize the sulphuric acid, whereupon the liquid was filtered and fractionated. The yield amounted to 90.8% of the theoretical yield of phenol and 89.7% of the theoretical yield of acetone.

EXAMPLE 2

The autoclave used in Example 1 was filled with 20 litres of a reaction mixture obtained in accordance with Example 1 before neutralization of the sulphuric acid.

The liquid in the autoclave was heated to 70° C. and an 85% solution of cumene hydro-peroxide in cumene was added till

the sulphuric acid concentration in the reaction mixture was 0.3%. At this stage sulphuric acid was added with the cumene hydro-peroxide, so as to maintain a 0.3% concentration of sulphuric acid in the reaction mixture.

The process was then continued as in Example 1. The yield amounted to 92% of phenol and 90.5% of acetone.

EXAMPLE 3

The autoclave used in Example 1 was filled with 20 litres of the reaction mixture obtained in accordance with Example 2 before neutralization of the sulphuric acid. The reaction mixture was heated to 50° C. and concentrated sulphuric acid was added till the concentration thereof reached 1%. At this stage, the addition of an 85% solution of cumene hydro-peroxide in cumene was started, further sulphuric acid being added in order to maintain the concentration steady at 1%.

The process was then continued as in Examples 1 and 2. The yield amounted to 91.5% of phenol and 90.2% of acetone.

Since the reaction mixture, when at least a 70% solution of cumene hydro-peroxide in cumene is employed, is homogeneous, the process may be carried out as a continuous operation. Continuous operation adds to the usefulness of the invention, for it results in considerable economic advantages.

WHAT WE CLAIM IS:—

1. Process for producing phenol and acetone by decomposing cumene hydroperoxide by means of concentrated sulphuric acid, characterised in that a solution of the cumene hydroperoxide in cumene at a concentration of at least 70% by weight and concentrated sulphuric acid are gradually added to cumene or to a previously decomposed solution of cumene hydroperoxide in cumene, the rate of addition being so controlled that the concentration of the acid in the reaction mixture is maintained in the range of from 0.1% to 1.1% by weight throughout the process.

2. Process as claimed in Claim 1, wherein the decomposition is carried out as a continuous operation.

3. Process as claimed in Claim 1 or 2, wherein the reaction temperature is in the range of from 40° C. to 100° C.

4. Process for producing phenol and acetone by decomposing cumene hydroperoxide, substantially as described in any one of the foregoing examples.

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